SYNTHESIS AND CHARACTERIZATION OF Sb_2O_3 NANOPARTICLES BY CHEMICAL REDUCTION METHOD

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by

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DECLARATION

I declare that this thesis is the result of my own research, that is does not incorporate without acknowledgement any material submitted for a degree or diploma in any university and does not contain any materials previously published, written or produced by another person except where due reference is made in the text.

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LIST OF ABBREVIATIONS

Al	:	Aluminum
APCVD	:	Atmospheric Pressure Chemical Vapor Deposition
Ar	:	Argon
B_2O_3	:	Boron Trioxide
Co	:	Cobalt
CO_2	:	Carbon Dioxide
CTAB	:	Cetyl Trimethyl Ammonium Bromide
Cu	:	Copper
EG	:	Ethylene Glycol
FCC	:	Face-Centered Cubic
Fe	:	Iron
H_2O	:	Water
HILH	:	Hybrid Induction and Laser Heating
HRTEM	:	High Resolution Transmission Electron Microscope
ICDD	:	International Centre for Diffraction Data
In_2O_3	:	Indium Trioxide
JCPDS	:	Joint Committee on Powder Diffraction Standard
LED	:	Light Emitting Device
MOCVD	:	Metal Organic Chemical Vapor Deposition
MoO_3	:	Molybdenum Oxide
N_2H_5OH	:	Hydrazine
NaOH	:	Sodium Hydroxide
Ni	:	Nickel

O_2	:	Oxygen
Pb:	:	Lead
PbO	:	Lead Oxide
PET	:	Poly(ethylene terephthalate)
PMMA	:	Poly(methyl methacrylate)
PVA	:	Polyvinyl Alcohol
RoHS	:	Restrictions of Hazardous Substances
SAED	:	Selected Area Electron Diffraction
Sb	:	Antimony
$SbCl_3$	:	Antimony Trichloride
SbO_2	:	Antimony Dioxide
Sb_2O_3	:	Antimony Trioxide
Sb_2O_4	:	Antimony Tetroxide
Sb_2O_5	:	Antimony Pentoxide
SDS	:	Sodium Dodecyl Sulfate
SEM	:	Scanning Electron Microscope
Sn	:	Tin
SnO_2	:	Tin Dioxide
TEM	:	Transmission Electron Microscope
TiO_2	:	Titanium Dioxide
UV-vis	:	Ultraviolet-visible
XRD	:	X-ray Diffraction
ZnO	:	Zinc Oxide

LIST OF SYMBOLS

^{o}C		Degree Centigrade
α		Alpha
β	:	Beta
γ	:	Gamma
λ	:	Lambda
θ	:	Angle
Å	:	Angstrom
a	:	Lattice Parameter
atm	:	atmosphere
Ci	:	curie
cos	:	cosinus
d	:	Interplanar Spacing
g	:	gram
g/cm^3	:	gram per cubic centimeter
g/mol	:	gram per mole
Gy	:	gray
h	:	hour
K	:	Kelvin
kV	:	kilovolt
M	:	Molarity
mA	:	milliampere
mg	:	milligram
min	:	minutes

ml	:	milliliter
mm	:	millimeter
mmol	:	millimoles
MPa	:	Megapascal
nm	:	nanometer
P^o	:	Vapor Pressure
Pa	:	Pascal
ppm	:	part per million
S/cm	:	Siemens per centimeter
wt	:	weight

LIST OF PUBLICATIONS

1. Chin, H. S., Cheong, K. Y. and Abdul Razak, K. (2010). Review on Oxides of Antimony Nanoparticles: Synthesis, Properties and Applications. Journal of Materials Science, 45, pp. 5993-6008. (Impact Factor: 1.471).

2. Chin, H.S., Cheong, K.Y. and Abdul Razak, K. (2011). Controlled Synthesis of Sb_2O_3 Nanoparticles by Chemical Reducing Method in Ethylene Glycol. Journal of Nanoparticle Research, 13, pp. 2807-2818. (Impact Factor: 2.478).

3. Chin, H.S., Cheong, K.Y. and Abdul Razak, K. (2011). Effect of Process Parameters on Size, Shape and Distribution of Sb_2O_3 Nanoparticles. Journal of Materials Science, 46, pp. 5129-5139. (Impact Factor: 1.471).

SINTESIS DAN PENCIRIAN NANOPARTIKEL Sb_2O_3 MELALUI KAEDAH PENURUNAN KIMIA

ABSTRAK

Nanopartikel antimoni trioksida (Sb_2O_3) dengan saiz kurang daripada 100 nm, berbentuk sfera dan taburan yang sekata telah berjaya dihasilkan melalui kaedah penurunan kimia. Antimoni triklorida ($SbCl_3$) telah diturunkan oleh hidrazin dalam kehadiran natrium hidroksida (NaOH) sebagai pemangkin dalam etilena glikol (EG) pada suhu 120 ^oC selama 60 minit. Bagi menghasilkan nanopartikel Sb_2O_3 dengan saiz partikel yang kecil (2 - 12 nm), berbentuk sfera dan taburan yang sekata, kesan kepekatan hidrazin ($[N_2H_5OH]/[SbCl_3]$ = 0.75, 5, 10, 20 dan 30), kepekatan NaOH ($[NaOH]/[SbCl_3]$ = 0, 1, 3 dan 5), kepekatan prapenanda ($[SbCl_3]/[N_2H_5OH]$ = 0.05, 0.1, 0.15 dan 0.2), suhu tindak balas (60, 90, 120 dan 150^oC), masa tindak balas (30, 60, 90 dan 120 minit) dan suhu didih (25, 50, 80 dan 110^oC) telah dikaji secara sistematik. Microskop penghantaran elektron (TEM), kawasan yang dipilih pola pembelauan elektron (SAED) dan mikroskop elektron resolusi tinggi (HRTEM) telah diaplikasikan untuk mengkaji morfologi dan penghabluran nanopartikel. Pemerhatian menunjukkan bahawa saiz partikel berkurang dan tidak berubah apabila kepekatan hidrazin ($[N_2H_5OH]/[SbCl_3]$) $\geq$ 10. Partikel yang lebih besar telah dihasilkan apabila kepekatan NaOH dan prapenanda, serta suhu dan masa tindak balas dinaikkan. Selanjutnya kajian penghabluran dan fasa nanopartikel telah dibantu oleh pembelauan sinar-X (XRD). XRD menunjukkan bahawa nanopartikel Sb_2O_3 adalah dalam fasa kubik. (ICDD file no. 00-043-1071) dengan kekisi jarak 1.68 Å. Walaubagaimanapun, puncak pembelauan $SbCl_3$ telah dikesan apabila hidrazin ditambahkan ke dalam campuran yang belum didih, campuran tersebut mengandungi

kedua-dua $SbCl_3$ dan NaOH dalam EG. Penambahan hidrazin ke dalam campuran yang belum mendidih mempengaruhi mekanisme penurunan $SbCl_3$ dan seterusnya penghasilan nanopartikel Sb_2O_3. Analisis ultraungu-nampak (UV-vis) spektrofotometer menunjukkan bahawa penyerapan panjang gelombang maksimum nanopartikel Sb_2O_3 telah berlaku dalam linkungan 280 hingga 318 nm. Keputusan kajian menunjukkan partikel yang kecil menyerap pada panjang gelombang UV-vis yang rendah, manakala partikel yang besar menyerap pada panjang gelombang UV-vis yang tinggi. Oleh itu, hubungan antara penyerapan panjang gelombang UV-vis nanopartikel dan saiznya telah ditetapkan.

SYNTHESIS AND CHARACTERIZATION OF Sb_2O_3 NANOPARTICLES BY CHEMICAL REDUCTION METHOD

ABSTRACT

Antimony trioxide (Sb_2O_3) nanoparticles with particle size less than 100 nm, spherical in shape and well distributed were successfully synthesized by chemical reducing method. Antimony trichloride ($SbCl_3$) was reduced by hydrazine in the presence of sodium hydroxide (NaOH) as catalyst in ethylene glycol (EG) at 120 ^oC for 60 minutes. In order to synthesis Sb_2O_3 nanoparticles with smaller particle size (2 - 12 nm), spherical in shape and well distribution, effects of hydrazine concentration ($[N_2H_5OH]/[SbCl_3]$ = 0.75, 5, 10, 20 and 30), NaOH concentration ($[NaOH]/[SbCl_3]$ = 0, 1, 3 and 5), precursor concentration ($[SbCl_3]/[N_2H_5OH]$ = 0.05, 0.1, 0.15 and 0.2), reaction temperature (60, 90, 120 and 150^oC), reaction time (30, 60, 90 and 120 minutes) and boiling temperature (25, 50, 80 and 110^oC) were investigated. Transmission electron microscope (TEM), selected area electron diffraction (SAED) pattern and high resolution electron microscope (HRTEM) were employed to study the morphology and crystallinity of the nanoparticles. It was observed that the particle size decreased and remained constant when concentration of hydrazine ($[N_2H_5OH]/[SbCl_3]$) $\geq$ 10. Increasing the concentration of NaOH and precursor, as well as reaction temperature and reaction time, larger particles were formed. Further study on the crystallinity and phase of the nanoparticles was assisted by X-ray diffraction (XRD). XRD revealed a cubic phase of Sb_2O_3 (ICDD file no. 00-043-1071) with lattice spacing of 1.68 Å. However, diffraction peaks of $SbCl_3$ were detected when hydrazine was added into an un-boiled mixture, which consists of both $SbCl_3$ and NaOH in EG. It was found that adding hydrazine to the un-boiled

mixture influenced the mechanism of reduction of $SbCl_3$ and eventually affected the production of Sb_2O_3 nanoparticles. From the ultraviolet-visible (UV-vis) spectrophotometer analysis, maximum absorption wavelengths of Sb_2O_3 nanoaparticles were occurred from 280 to 318 nm. The results showed that smaller particles were showed lower UV-vis absorption wavelength, while larger particles were showed higher UV-vis absorption wavelength. Therefore, correlation between UV-vis absorption wavelengths of the nanoparticles and their sizes has been established.